**ORIGINAL PAPER** 



# Geomechanical Properties of Clay Stabilised with Fly Ash-Based Geopolymer Subjected to Long-Term Sulfate Attack

Hayder H. Abdullah<sup>1</sup> · Mohamed A. Shahin<sup>1</sup>

Received: 5 June 2023 / Accepted: 11 October 2023 / Published online: 30 October 2023 © The Author(s) 2023

#### Abstract

Fly ash-based geopolymers have emerged as an eco-friendly alternative binder compared to conventional Portland cement for soil stabilisation. However, the gap in the current literature is the lack of a comprehensive study regarding the geomechanical behaviour of fly ash geopolymer-treated clay subjected to long-term sulfate attack, particularly in terms of potential ettringite formation and the corresponding impact on cementitious soil structure. The goal of this paper is to address this knowledge gap and provide a comprehensive study to fulfil it. In this work, sulfate attack was simulated by submerging geopolymer-treated clay specimens in two distinct sulfate-based solutions (i.e., sodium and magnesium), for one year. Subsequently, comparative analyses of the geomechanical and microstructural changes in geopolymer-treated clay under various curing conditions were conducted through unconfined compressive strength, direct shear, volume change and microscopic tests. The findings indicate that the addition of geopolymer for the stabilisation of clay soil significantly improves soil strength without affecting the soil volumetric response. Although the clay used exhibited similar qualitative stress–strain behaviour across all simulated attacks, notable quantitative differences emerged in the peak strength, stiffness and shear strength parameters. Such discrepancies can primarily be attributed to the varying buffering capacities (i.e., pH changes associated with acidification) of the sulfate solutions and the subsequent residual pH, cementitious product formation and strength enhancement within the treated clay.

Keywords Geopolymer · Soil treatment · Fly ash · Sulfate attack

# Introduction

Chemical treatment via deep mixing represents an effective technique for ground improvement, which typically involves mixing the soil with a binder (commonly Ordinary Portland cement) and water to create a stabilised soil column with an enhanced mechanical property [1-3]. However, the effectiveness of this technique is often compromised, particularly in coastal regions, due to the susceptibility of cement-treated soils to sulfate attacks. Such attacks stimulate the formation of extensive ettringite, resulting in soil structural decay [4, 5]. The formation of ettringite in cement-treated soils is a consequence of the interaction between calcium and alumina (byproducts of cement hydration) with sulfate present in

 Hayder H. Abdullah hayderhasan.abdullah@curtin.edu.au Mohamed A. Shahin

m.shahin@curtin.edu.au

<sup>1</sup> School of Civil and Mechanical Engineering, Curtin University, Perth, WA, Australia the soil or groundwater. The presence of clay in soils could potentially amplify this issue because clay minerals present an additional source of alumina that can combine with sulfate due to the high alkalinity of hydrated cement. The resultant volumetric changes of such soils could prompt the swelling and severe cracking of the cementitious soil structure [6]. Consequently, recognising the deleterious implications of ettringite on cemented soils, sulfate-resistant cement has been employed to stabilise soils under threat from sulfate attacks. Yet, the continuous use of sulfate-resistant cement might magnify the environmental consequences associated with cement production, including heightened CO<sub>2</sub> emissions [7], substantial energy consumption [8], raw material depletion [7] and significant landscape deterioration [8, 9]. The above concerns underscore the need for innovative solutions that can effectively stabilise sulfate-affected soils while concurrently mitigating the environmental impact.

Geopolymer, an emerging chemical binder, presents a promising solution for soil stabilisation [10-17]. This binder is synthesized from high-aluminosilicate materials with low calcium content (e.g., fly ash or metakaolin) which are

subjected to activation using an alkaline solution. Fly ash is preferable for geopolymer synthesis, given its availability as a coal power plant by-product [18, 19]. The improvement of the mechanical properties in treated soil utilizing fly ash-based geopolymer, including strength and durability, is credited to the formation of artificial bonds between soil particles through geopolymerisation. This process entails the following processes: (1) breakdown of aluminosilicate oxides present in fly ash by the high concentration of hydroxyl ions (OH-) in the alkaline activator; (2) diffusion of dissolved aluminium and silicon complexes; (3) gel formation by the chemical condensation process that occurs between the residual alkali cations, which could be sodium (Na+) or potassium (K+), and the aluminium and silicon complexes.; and (4) gel hardening among soil particles, which ultimately enhancing soil properties. Sodium Aluminium Silicate Hydrate (N–A–S–H) constitutes the primary chemical structure of the final product of geopolymerisation. To optimize the performance of geopolymer within treated soil, calcium-containing additives (e.g., ground granulated blast-furnace slag) are usually utilised to influence the formation of auxiliary Calcium Aluminium Silicate Hydrate C-A-S-H products to promote high mechanical properties of soil when mixed with geopolymer at ambient temperature [20, 21]. However, the presence of calcium in the geopolymer-soil matrix might trigger ettringite formation when sulfate is present, potentially undermining the mechanical properties. Research focusing on the long-term effects (>1 year) of sulfate attack on clay treated with fly ash-based geopolymer that incorporates blast-furnace slag is quite limited. Most existing studies predominantly examine the effects of sulfate on treated clay after brief curing periods of 7, 28, and 90 days, typically using a specific mix [12, 22, 23]. These studies often place a greater focus on surface mixing rather than deep mixing.

The present study seeks to explore the geomechanical behaviour of fly ash-based geopolymer-treated clay incorporating slag when subjected to long-term sulfate attacks. This was achieved by immersing 28-day cured treated kaolin clay samples with fly ash-based geopolymer incorporating slag in two different sulfate-rich solutions, as well as tap water, for one year. The investigation focused on determining geomechanical properties such as stress-strain behaviour, peak strength and modulus of elasticity through unconfined compression tests. Additionally, the friction angle ( $\phi$ ') and cohesion  $(\acute{c})$  of the stabilised clay were assessed using direct shear tests. Furthermore, scanning electron microscopy (SEM) was employed to gain a deeper understanding of the stabilisation processes and potential formation of ettringite in the clay treated with geopolymer. This study aims to broaden our knowledge of how geopolymer-treated clay responds to sulfate attacks, a crucial factor in encouraging its broader application in the field of soil stabilisation.

## **Materials and Methodology**

### Clay

Commercially available kaolin clay was used for this study. The clay used was characterised by a plastic limit (*PL*) of 27%, a liquid limit (*LL*) of 53% with 99% passing the 75  $\mu$ m sieve and a clay fraction (<2  $\mu$ m) of 79.4% (see Table 1). The pH value and activity index of the clay used were 7.8 and 0.33, respectively. The Australian Standards were strictly adhered to during the execution of all tests. This included the particle size distribution [24], Atterberg limits [25, 26], and pH tests [27]. The kaolin clay employed was identified as high-plasticity (CH) clay as per the Unified Soil Classification System (USCS).

### **Components of Geopolymer**

The geopolymer utilised in this study consists of fly ash and an alkaline activator. The used fly ash, classified as Class F according to AS 3582.1 1998, was obtained from the Gladstone Power Station. Its selection was influenced by its rich aluminosilicate content and its proven track record in geopolymer applications [7, 19]. Numerous researchers have recommended the utilization of sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>) and sodium hydroxide (NaOH) as the alkaline activator [13, 14, 16, 28, 29]. PQ Australia provided the sodium silicate solution, containing 14.7% Na<sub>2</sub>O and a mass ratio (SiO<sub>2</sub>/Na<sub>2</sub>O) of 2. Sodium hydroxide pellets were dissolved in deionised water, achieving a concentration of 14 molars. A weight ratio of 2.33 comprising sodium silicate to sodium hydroxide was employed to optimize the geopolymer reaction phase, following the suggestion by Hardjito [19]. To improve the performance of the used fly ash-based geopolymer at ambient temperature, ground granulated blast furnace slag (GGBFS), was incorporated [20, 21]; this was obtained from Independent

Table 1 Properties and classification utilized clay

Property	Value/ designa- tion
Liquid limit, LL (%)	53
Plasticity index, PI (%)	26
Particles < 75 µm (%)	99
Particles < 2 µm (%)	79.4
pH value	7.8
Soil classification (USCS)	СН

USCS Unified soil classification system

Material	Chemica	al composit	ion (%)						
	Na <sub>2</sub> O	MgO	Fe <sub>2</sub> O <sub>3</sub>	CaO	$Al_2O_3$	K <sub>2</sub> O	SiO <sub>2</sub>	SO <sub>3</sub>	LOI
GGBFS	0.31	5.99	0.52	45.45	12.25	0.38	29.96	3.62	2.39
Fly ash	0.77	1.45	12.48	4.30	25.56	0.70	51.11	0.24	0.57
	Material GGBFS Fly ash	$\begin{tabular}{ c c c c } \hline Material & Chemic: & & & \\ \hline \hline Na_2O & & & \\ \hline GGBFS & 0.31 & & \\ Fly ash & 0.77 & & \\ \hline \end{tabular}$	$\begin{tabular}{ c c c c } \hline Material & Chemical composit\\ \hline \hline Na_2O & MgO \\ \hline \hline GGBFS & 0.31 & 5.99 \\ \hline Fly ash & 0.77 & 1.45 \\ \hline \end{tabular}$	$\begin{tabular}{ c c c c c } \hline Material & Chemical composition (\%) \\ \hline \hline Na_2O & MgO & Fe_2O_3 \\ \hline GGBFS & 0.31 & 5.99 & 0.52 \\ Fly ash & 0.77 & 1.45 & 12.48 \\ \hline \end{tabular}$	$\begin{tabular}{ c c c c c c c c c c c c c c c c c c c$	$\begin{tabular}{ c c c c c c c c c c c c c c c c c c c$	$\begin{tabular}{ c c c c c c c c c c c c c c c c c c c$	$\begin{tabular}{ c c c c c c c c c c c c c c c c c c c$	$\begin{tabular}{ c c c c c c c c c c c c c c c c c c c$

LOI loss of ignitions

Cement & Lime Limited. Table 2 presents the chemical compositions of the employed GGBFS and fly ash.

# Soil-Geopolymer Admixtures and Specimen Preparation

In this investigation, the mixing process was designed to replicate the in-situ wet mixing technique employed in Deep Soil Mixing (DSM) applications. Initially, water was combined with dry clay to reach a liquid limit state, followed by a curing period of at least 24 h in sealed containers. Subsequently, the clay was blended with 10% and 20% by weight of a pre-prepared geopolymer mix containing a slag/fly ash ratio of 20% by weight, based on research conducted by Abdullah, et al. [30] that aimed to enhance short-term geopolymer hardening under ambient curing conditions for stabilising kaolin clay. The concentration of the liquid activator was kept constant with a ratio of [activator/(fly ash+slag)] of 1.0 to maximise the reaction [10]. It's important to note that the percentages of 10% and 20% for geopolymer contents represent the ratio of dry additive to the total weight of dry clay before activation.

After the mixing process, the treated clay was remoulded using a split cylindrical PVC mould, measuring 50mm in diameter and 100mm in height. This was done to achieve a diameter-to-height aspect ratio of 1:2, typically needed for UCS testing. Additional sets of specimens with  $(60 \times 60 \times 30 \text{ mm})$  size were also prepared to comply with the requirements of direct shear tests. Direct tapping was employed on the mould sides to manage entrapped air in all specimens; micro air voids were unavoidable due to workability and related viscosity of mixtures considering the designated activator and water content at the liquid limit state (see Fig. 1). To prevent any interference caused by the hardening of the stabiliser, attention was given to ensure the sampling process was finished within 30 min post-mixing.

## Sulfate Attack and Curing

Initially, all samples underwent a curing process for 28 days at 60% humidity and temperatures ranging from 18 to 22 °C. Following this, they were subjected to additional curing under sulfate attack for a period of up to 365 days. The sulfate attack in geopolymer-treated used clay was simulated by submerging the initially cured specimens in sulfaterich solutions of 50 g/L concentration [31] for 90 days and 365 days. In this study, two different types of sulfate, i.e., sodium sulfate ( $Na_2SO_4$ ) and magnesium sulfate ( $MgSO_4$ ), were used to allow studying the effect of different sulfate types on treated clay, as suggested by many researchers e.g., [5, 32–34]. Care was taken to replace sodium and magnesium sulfate solutions every 60 days to ensure a constant concentration of sulfate during the designated sulfate attack period. An additional set of specimens were also submerged in water for 90 days and 365 days. These samples were tested for comparison with the geopolymer-stabilized samples subjected to sulfate attack. Different curing regimes of the geopolymer-treated clay used are provided in Table 3.



Fig. 1 Geopolymer-treated clay specimens

International Jour	nal of Geosynthetic	cs and Ground End	aineerina (2023) 9:75
			<b>J</b>

Table 3	Details	of sampl	es used	under	different	curing	regimes
---------	---------	----------	---------	-------	-----------	--------	---------

Geo- polymer (%)	Curing regime (days)							
	Initial curing (ambient tempera- ture18–22 °C)	Sodium sulfate solu- tion	Magnesium sulfate solu- tion	Water				
10	28	90	90	90				
		365	365	365				
20	28	90	90	90				
		365	365	365				

# **Tests Conducted**

UCS tests were performed to investigate the compressive strength of treated clay samples under different curing conditions, as outlined in Table 3. The experiments were conducted in compliance with the AS 5101.4 Australian Standards [35], utilizing a strain rate of 1% per minute. Tests were performed on cylindrical samples after 90 days and 365 days of curing. During each interval, two samples were examined, and the average outcome was considered. In instances where the UCS values deviated significantly (>20%), a third specimen was conducted for accuracy and the outlier was dismissed.

Direct shear tests were utilized to gain an understanding of the shear strength of treated clay under varying curing conditions. Treated specimens, with dimensions of (60 mm×60 mm×30 mm), were first subjected to sulfate attacks for 365 days before applying the test conditions following AS 1289.6.2.2 [36]. Normal stresses of 20 kPa, 40 kPa and 80 kPa, simulating average field effective stresses for shallow soil elements, were applied on specimens and then sheared along a pre-determined horizontal plane between the two halves of the shear box at a fixed displacement rate of 0.5mm/min. A minimum of three specimens for the selected mixture/curing period were tested to enable the prediction of the Mohr-Coloumb failure envelope and determination of the geomechanical properties, friction angle ( $\phi$ ') and cohesion ( $\hat{c}$ ).

Dimensions of the cured cylindrical specimens were monitored after the conclusion of the maximum designated curing period (i.e., 365 days) to determine the total volume change within treated specimens. To accurately determine the volume change of the treated clay before and after immersion, the average volume change of two specimens was taken. Before treatment, Scanning Electron Microscopy (SEM) was utilised for analysing the microstructure of the soil, while post-treatment observations using SEM aimed at identifying binder-induced alterations and sulfate-induced deterioration. This process was conducted by employing MIRA3 XMU, a fully PC-controlled apparatus.

## **Results and Discussions**

#### **UCS Stress–Strain Performance**

Figure 2 presents the UCS test results for clay treated with 10% and 20% geopolymer, which was cured for durations of 90 days and 365 days. The different curing conditions include water, sodium sulfate, and magnesium sulfate solutions. Regardless of the curing process used, it was observed that geopolymer-treated clay with 10% geopolymer content that was cured for 90 days (shown in Fig. 2a) demonstrated a predominantly brittle yield stress-strain response. This involved the stress reaching a maximum value before failure, a contrast to the untreated clay at its liquid limit state, which exhibited zero stress-strain response. At 365 days of curing, the 10% geopolymer addition showed an increase in the peak UCS value and a further reduction in the corresponding strain, indicating a higher brittle stress-strain response with the increase in curing time. Similar qualitative behaviour was also observed for the 20% geopolymer addition with a more prominent brittle response (Fig. 2b) and for both curing times of 90 days and 365 days.

The quantitative improvement in the peak UCS and stiffness, represented by the secant modulus of elasticity at 50% maximum stress  $(E_{50})$ , along with a pictorial representation of the brittle failure mode of treated specimens are presented in Figs. 3, 4, 5. In all cases, the enhanced strength response of the treated clay for different curing conditions can be ascribed to the formation of the artificial cementation (N-A-S-H) products within the clay and the associated enhancement in clay structure due to geopolymer addition. as explained by Phummiphan et al. [13]. As the geopolymer content increases, the modified structure of the treated clay yields a higher quantity of artificial cementitious products. Consequently, there is a significant enhancement in the strength performance of the specimens [13, 37–39]. Moreover, the increase in curing time for a specific geopolymer content allowed more time for the geopolymerisation reaction to produce more cementitious products and enhance the stress-strain response of the stabilised clay. The increase in curing time gradually reduced the rates of strength gain and stiffness in geopolymer-treated clay, irrespective of the specific geopolymer content, indicating the rates were not constant. This can be detected from the variation of the peak UCS and  $E_{50}$  with the curing time (see Figs. 3 and 4). The decrease in the improvement of the mentioned soil properties over time can be linked to the gradual deceleration of the geopolymerisation reaction. This slowdown is a result of the depletion of various elements in the reaction environment throughout the curing process. However, the quantitative influence of geopolymer addition at different curing times is unique for each curing method. Figure 3 demonstrates that



Fig. 2 Typical stress-strain response of geopolymer-treated kaolin clay cured with water and sulfate solutions and over different curing durations of 90 and 365 days: a 10% geopolymer; and b 20% geopolymer

the peak UCS of specimens cured in water increased by 260 kPa and 352 kPa after 90 days and 365 days, respectively, with the addition of 10% geopolymer. Meanwhile, curing in sodium sulfate and magnesium sulfate solutions enhanced the strength by 212 kPa and 227 kPa at 90 days, and 280 kPa and 343 kPa at 365 days for the same geopolymer content. The variable impact of the curing process on the peak UCS of geopolymer-treated clay remained consistent for the 20% geopolymer composition, across both 90-day and 365-day curing durations. The difference in the UCS peak response to geopolymer treatment between different curing conditions is also evident from the quantitative change in stiffness (i.e.,  $E_{50}$ ), as depicted in Fig. 4. The findings suggest that specimens treated with geopolymer and cured in sulfate solutions demonstrated lower strength response compared to those cured in water. This might be explained by the possible formation of expansive ettringite within geopolymer-treated clay and its negative impact on soil structure [12, 22, 40].

Figures 3 and 4 also indicate that specimens cured in magnesium sulfate solutions exhibit the least reaction to geopolymer treatment when compared to those cured in sodium

sulfate solution and water alone. This result confirms the negative role of specific types of sulfates on the effectiveness of clay stabilisation with fly ash-based geopolymer incorporating slag. The findings resemble those described by Yi et al. [23], which were reportedly associated with swelling and cracking resulting from inconsistent formation of ettringite in treated clay samples cured for 90 days at different sulfate solutions. However, the varying effects of different sulfate solutions on geopolymer stabilisation may not only impact the creation of ettringite within treated clay but could also potentially alter the pH value of the curing solution and the residual pH of the treated clay samples necessary to maintain geopolymerisation reaction. The possible effect of the mentioned factors will be investigated later in this work.

#### Shear Strength Performance—Direct Shear Test

The peak failure envelopes for 10% geopolymer content cured for 90 days and 365 days in tap water, sodium sulfate solution, and magnesium sulfate solution are depicted in Fig. 6. These results correspond to the various normal



Fig. 3 Peak strength response of geopolymer-treated kaolin clay cured with water and sulfate solutions and over different curing durations of 90 and 365 days: **a** 10% geopolymer; and **b** 20% geopolymer



Fig. 4 Effect of sodium and magnesium solutions on stiffness gain of geopolymer-treated kaolin clay at 90 and 365 days: **a** 10% geopolymer; and **b** 20% geopolymer

stresses (i.e., 20 kPa, 40 kPa, and 80 kPa) utilized in the direct shear tests. It is important to mention that the mechanical properties of 20% geopolymer-treated clay could not be examined due to its strength exceeding the capacity of the direct shear machine and thus was excluded.

Regardless of the duration of curing, a linear failure envelope can represent the peak shear strength of clay treated with 10% geopolymer in the  $\sigma_n - \tau'$  space under varying curing conditions, as shown in Fig. 6. The capability of geopolymer treatment underwater immersion to shift the stress states beyond the failure envelope of untreated clay mixed at the liquid limit state, which is a zero-strength state, is clearly visible. This consistently results in a failure envelope for geopolymer treatment that surpasses that of untreated clay. When the curing time for the 10% treated clay is extended from 90 to 365 days, both the gradient and  $\sigma_n$ -axis intercept values of the envelope exhibit a rise. This suggests that the effective shear strength parameters, namely effective cohesion (c) and friction angle ( $\phi$ ), also increase for the treated clay, as illustrated in Fig. 7. The enhancement in the effective cohesion of the treated clay can be attributed to the augmented cementation that results from the continuing geopolymerisation process. This bears a resemblance to the effect of curing time on the UCS performance mentioned previously. Likewise, the enhanced effective friction angle in these specimens is attributable to the presence of sizeable particles generated by the clay's bonded clusters after stabilisation [41]. However, the superimposition of  $\sigma_n - \tau'$  slopes in Fig. 6 and the corresponding effective shear strength parameters (Fig. 7) significantly demonstrate the impact of sulfate on the stress failure envelope response of geopolymer-treated clay. Particularly, curing in magnesium sulfate solution shows the lowest strength response to the treatment with geopolymer, where increases for 10% geopolymer specimens were only 0.81 and 0.74 folds for  $\dot{c}$  and  $\phi$ , respectively, compared to the values obtained for curing in tap water at 365 days of curing.

#### **Volumetric Changes**

The volumetric changes in the geopolymer-treated clay specimens were examined, with findings illustrated in Table 4, corresponding to various curing techniques such as water, sulfate, and magnesium solutions. Irrespective of the curing method, the 10% and 20% geopolymer specimens displayed substantial resistance to the volumetric changes throughout the long-term curing period of 365 days. As can be seen in Table 4, specimens that underwent curing in sodium sulfate solution demonstrated the poorest performance, marked by the most significant changes. However, the overall maximum volumetric variation was negligible, falling below 1.2% for specimens with 10% geopolymer content and 0.8% for those with

**Fig. 5** Typical influence of geopolymer addition on Unconfined Compressive Strength (UCS) failure mode of clay treated with a 20% geopolymer content, and cured for a year under different conditions: **a** water; **b** sodium sulfate solution; and **c** magnesium sulfate solution





**Fig. 6** Peak shear failure envelops corresponding to 20, 40, and 80 kPa normal stresses for kaolin clay stabilised with 10% geopolymer content and cured for 90 and 365 days in water, sodium sulfate solution and magnesium sulfate solution

20% geopolymer content. As noted by Pedarla, et al. [42], this is significantly lower than the 10% threshold typically used to denote volumetric stability in treated soils. The



**Fig. 7** Effective shear strength parameters for kaolin clay stabilised with 10% geopolymer, and cured for 90 and 365 days in water, sodium sulfate solution and magnesium sulfate solution

Table 4	Volumetric	changes	for	geopolymer-treated	specimens	sub-
jected to	sulfate atta	ck at 365	day	s of curing		

Curing liquid	Volumetric change (%)				
	10% Geopolymer	20% Geopol- ymer			
Water	0.90	0.50			
Sodium sulfate	1.20	0.80			
Magnesium sulfate	0.98	0.70			

insignificant stable volumetric performance of treated specimens subjected to sulfate curing indicates no significant formation of any expansive products within the treated soil particles indicating no formation of ettringite and this will be confirmed below by the microstructural analysis.

# **Microstructure of Treated Kaolin Clay**

The SEM images of both untreated and 20% geopolymertreated clay, which were fully submerged in water, sodium sulfate, and magnesium sulfate solutions for 365 days, are presented in Fig. 8. In Fig. 8a, the unsoaked kaolin clay's microstructure primarily comprises plate-like particles with a significant degree of micro-porosity. Compared to untreated clay, the geopolymer-treated soil particles submerged in different liquids (Fig. 8b-d) are tightly packed and encapsulated by the N-A-S-H products; no expansive products were detected within the treated soil particles indicating no formation of ettringite. Such observation contradicts the findings shown by Yi et al. [23], where the formation of ettringite was detected in geopolymer-treated clay submerged in sulfate solution. However, the degree of packing of soil particles within treated clay specimens seems to vary with the change of curing solution and it seems to be minimised for specimens cured in the magnesium sulfate



Fig. 8 SEM images of untreated and geopolymer-treated clay cured for 365 days: a untreated clay; b submerged in water; c submerged in sodium sulfate solution; and d submerged in magnesium sulfate solution solution (Fig. 8c). Such observation confirms a direct impact of the type of curing solution on the geopolymerisation process and precipitation of the N-A-S-H products within treated soil particles. Such impact might be explained by the effect of curing solution type on the residual pH value required to maintain the reaction rate within treated clay along the curing period. It has been found in the literature that a minimum pH value of 12 is necessary to maintain the geopolymerisation reaction in treated clays [43]. The high level of pH induced by the activator within the geopolymer seems to reduce at different rates using different curing solutions. Such variable impact on the pH of the stabilised clay can be justified by the differences in buffering capacity of different curing solutions, including water and their distinct abilities to absorb, retain and release ions, such as hydroxide (OH-) ions. Sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>) and magnesium sulfate (MgSO<sub>4</sub>) are considered neutral salts since they are formed by the reaction between a strong acid  $(H_2SO_4)$  and a strong base (NaOH) or moderately strong base  $(Mg(OH)_2)$ . Under normal conditions, neutral salts do not alter the pH of a solution significantly. As such, their aqueous solutions are expected to have a pH value near 7, which is considered neutral. However, due to the higher charge of magnesium ion (Mg<sup>2+</sup>) compared to the sodium ion (Na<sup>+</sup>), there might be a slight difference in their pH values. The magnesium ion (Mg<sup>2+</sup>) can slightly hydrolyze water molecules, forming  $Mg(OH)_2$  and thus releasing H<sup>+</sup> ions into the solution, slightly decreasing the pH value. Consequently, the pH of both sodium sulfate and magnesium sulfate solutions at 50 g/l concentration will be near 7, with the magnesium sulfate solution potentially having a slightly lower pH due to negligible hydrolysis. To investigate the effect of different sulfates on geopolymer treatment, the residual pH level of 20% geopolymer specimens cured in differing liquids was examined after 365 days of curing. For specimens cured in tap water, the pH value was 11.2, whereas, for sodium sulfate and magnesium sulfate solutions at a selected concentration of 50 g/l used in this study, the pH values of specimens show residual values of 10.6 and 10.1, respectively. The different sulfate solutions seem to partially impact the pH within the treated clay specimens at different rates causing variable performance of geopolymer within treated soil compared to controlled specimens cured in water only.

# Conclusions

This study examined the mechanical performance of a specific geopolymer mixture for stabilising clay soils in deep mix applications subjected to long-term sulfate attack. Various experimental tests, including unconfined compressive strength, direct shear test, volume changes and microstructural analysis, were carried out on kaolin clay treated with geopolymer cured with sodium sulfate, magnesium sulfate and tap water for one year. The key findings of the study are encapsulated in the following points:

- The compressive strength of the treated clay was profoundly influenced by the geopolymer content and curing time. Both variables contributed to an increase in yield strength, stiffness, cohesion, and angle of internal friction.
- It was interestingly noted that the presence or type of sulfate did not notably influence the volumetric changes of the geopolymer-treated clay. This finding points to an absence of expansive product formation, which was corroborated by the scanning electron microscope (SEM) analysis.
- All clay soil specimens displayed an improvement in strength when subjected to curing in both sulfate solutions and tap water, however, the magnitude of this improvement varied significantly.
- The type of sulfate was identified as a crucial factor in determining the strength characteristics of geopolymertreated clay. Specimens attacked by magnesium sulfate showed a lower stress-strain response when compared to those cured in sodium sulfate solution and tap water.
- The variation between sulfate types was primarily ascribed to the differences in buffering capacity, ion absorption, and residual pH value within the specimens.

In conclusion, the study findings indicate that fly ashbased geopolymer is an effective binder for clay stabilisation in deep mix applications, specifically those subjected to sulfate attack. However, there is still a compelling need for additional studies on geopolymers sourced from diverse materials, including waste and industrial byproducts, with the intent of stabilising sulfate-rich natural soils. Such prospective investigation may pave the way for the creation of cost-efficient solutions, specifically tailored for soils rich in sulfate.

Author Contributions HHA and MAS were both involved in conceptualisation, investigation, preparing the original draft, reviewing and editing the manuscript, supervision, and project administration. HHA also managed the methodology and formal analysis. Both authors have reviewed and given their consent to the final version of the manuscript to be published.

**Funding** Open Access funding enabled and organized by CAUL and its Member Institutions.

**Data Availability** The datasets generated during and/or analysed during the current study are not publicly available but are available from the corresponding author on reasonable request.

#### **Declarations**

**Conflict of Interest** The authors declare that there are no potential conflicts of interest concerning the research, authorship, and/or publication of this study.

**Open Access** This article is licensed under a Creative Commons Attribution 4.0 International License, which permits use, sharing, adaptation, distribution and reproduction in any medium or format, as long as you give appropriate credit to the original author(s) and the source, provide a link to the Creative Commons licence, and indicate if changes were made. The images or other third party material in this article are included in the article's Creative Commons licence, unless indicated otherwise in a credit line to the material. If material is not included in the article's Creative Commons licence and your intended use is not permitted by statutory regulation or exceeds the permitted use, you will need to obtain permission directly from the copyright holder. To view a copy of this licence, visit http://creativecommons.org/licenses/by/4.0/.

# References

- 1. Nicholson PG (2014) Soil improvement and ground modification methods. Butterworth-Heinemann
- 2. Kitazume M (2013) The deep mixing method/Masaki Kitazume, Masaaki Terashi. CRC Press, Boca Raton
- Pourakbar S, Huat BK (2017) A review of alternatives traditional cementitious binders for engineering improvement of soils. Int J Geotech Eng 11:206–216
- Helson O, Eslami J, Beaucour A-L, Noumowe A, Gotteland P (2018) Durability of soil mix material subjected to wetting/ drying cycles and external sulfate attacks. Constr Build Mater 192:416–428
- Atashband S, Sabermahani M, Elahi H (2021) Internal and external effects of sodium sulfate on the strength of soil cement. Iran J Sci Technol Trans Civ Eng 45:2595–2610
- Rajasekaran G (2005) Sulphate attack and ettringite formation in the lime and cement stabilized marine clays. Ocean Eng 32:1133–1159
- Garcia-Lodeiro I, Palomo A, Ernandez-Jimenez AF (2014) An overview of the chemistry of alkali-activated cement-based binders. In: Pacheco-Torgal F, Labrincha J, Leonelli C, Sargent P (eds) Handbook of alkali-activated cements, mortars and concretes. Elsevier Science: Instituto Eduardo Torroja (IETcc-CSIC), Madrid, pp 20–42
- Sargent P (2014) The development of alkali-activated mixtures for soil stabilisation. In: Pacheco-Torgal F, Labrincha J, Leonelli C, Sargent P (eds) Handbook of alkali-activated cements, mortars and concrete. Elsevier Science, Kent
- 9. Tayibi H, Choura M, López FA, Alguacil FJ, López-Delgado A (2009) Environmental impact and management of phosphogypsum. J Environ Manag 90:2377–2386
- Abdullah HH, Shahin MA (2022) Geomechanical behaviour of clay stabilised with fly-ash-based geopolymer for deep mixing. Geosciences 12:207
- Zhang M, Guo H, El-Korchi T, Zhang G, Tao M (2013) Experimental feasibility study of geopolymer as the next-generation soil stabilizer. Constr Build Mater 47:1468–1478
- Zhang M, Zhao M, Zhang G, Nowak P, Coen A, Tao M (2015) Calcium-free geopolymer as a stabilizer for sulfate-rich soils. Appl Clay Sci 108:199–207
- Phummiphan I, Horpibulsuk S, Sukmak P, Chinkulkijniwat A, Arulrajah A, Shen S-L (2016) Stabilisation of marginal lateritic soil using high calcium fly ash-based geopolymer. Road Mater Pavement Des 0629:1–15

- Sargent P, Hughes PN, Rouainia M, White ML (2013) The use of alkali activated waste binders in enhancing the mechanical properties and durability of soft alluvial soils. Eng Geol 152:96–108
- Phetchuay C, Horpibulsuk S, Arulrajah A, Suksiripattanapong C, Udomchai A (2016) Strength development in soft marine clay stabilized by fly ash and calcium carbide residue based geopolymer. Appl Clay Sci 127:134–142
- Cristelo N, Glendinning S, Fernandes L, Pinto AT (2013) Effects of alkaline-activated fly ash and Portland cement on soft soil stabilisation. Acta Geotech 8:395–405
- 17. Yaghoubi M, Arulrajah A, Disfani MM, Horpibulsuk S, Bo MW, Darmawan S (2018) Effects of industrial by-product based geopolymers on the strength development of a soft soil. Soils Found 58:716–728
- Gianoncelli A, Zacco A, Struis RP, Borgese L, Depero LE, Bontempi E (2013) Fly ash pollutants, treatment and recycling. Pollutant diseases, remediation and recycling; environmental chemistry for a sustainable world, vol 4. Springer, pp 103–213
- Hardjito D (2005) Studies on fly ash-based geopolymer concrete. Ph.D. thesis, Curtin University of Technology, Perth, WA, Australia
- Yip CK, Lukey G, Van Deventer J (2005) The coexistence of geopolymeric gel and calcium silicate hydrate at the early stage of alkaline activation. Cem Concr Res 35:1688–1697
- Granizo ML, Alonso S, Blanco-Varela MT, Palomo A (2002) Alkaline activation of metakaolin: effect of calcium hydroxide in the products of reaction. J Am Ceram Soc 85:225–231
- 22. Khadka SD, Jayawickrama PW, Senadheera S, Segvic B (2020) Stabilization of highly expansive soils containing sulfate using metakaolin and fly ash based geopolymer modified with lime and gypsum. Transport Geotech 23:100327
- 23. Yi X, Wang G, Zhang B, Zhang G, Liu Y, Luo Z (2023) Durability deterioration of geopolymer stabilized soft soil under sodium sulfate and magnesium sulfate attack: a comparative study. Buildings 13:1075
- 24. Standards Australia (2009) Methods of testing soils for engineering purposes-Method 3.6.1: soil classification tests-determination of the particle size distribution of a soil-standard method of analysis by sieving, 1–12
- Standards Australia (2009) Methods of testing soils for engineering purposes-method 3.2.1: soil classification tests-determination of the plastic limit of a soil-standard method, 1–5
- Standards Australia (2009) Method 3.2.1: soil classification tests-determination of the plastic limit of a soil-standard method, 1–5
- 27. Standards Australia (1997) Methods of testing soils for engineering purposes-Method 4.3.1: soil chemical tests-determination of the pH value of a soil-electrometric method, 1–5
- Rios S, Cristelo N, Viana-Da-Fonseca A, Ferreira C (2016) Structural performance of alkali-activated soil ash versus soil cement. J Mater Civ Eng 28:0001398
- Verdolotti L, Iannace S, Lavorgna M, Lamanna R (2008) Geopolymerization reaction to consolidate incoherent pozzolanic soil. J Mater Sci 43:865–873
- Abdullah H, Shahin MA, Sarker P (2019) Use of fly-ash geopolymer incorporating ground granulated slag for stabilisation of kaolin clay cured at ambient temperature. Geotech Geol Eng 37:721–740
- 31. American Society for Testing and Materials (2012) Length change of hydraulic-cement mortars exposed to a sulfate solution
- Singhi B, Laskar AI, Ahmed MA (2017) Mechanical behavior and sulfate resistance of alkali activated stabilized clayey soil. Geotech Geol Eng 35:1907–1920

- Yi Y, Li C, Liu S, Al-Tabbaa A (2014) Resistance of MgO-GGBS and CS-GGBS stabilised marine soft clays to sodium sulfate attack. Géotechnique 64:673–679
- Yi Y, Li C, Liu S, Jin F (2015) Magnesium sulfate attack on clays stabilised by carbide slag- and magnesia-ground granulated blast furnace slag. Géotech Lett 5:306–312
- 35. Standards Australia (2008) Methods for preparation and testing of stabilized materials -Method 4: unconfined compressive strength of compacted materials, 1–13
- 36. Standards Australia (2020) Methods of testing soils for engineering purposes - Method 6.2.2: Soil strength and consolidation tests—determination of shear strength of a soil - Direct shear test using a shear box, 1–17
- Cristelo N, Glendinning S, Pintio A, Fernandes L (2012) Effect of calcium content on soil stabilisation with alkaline activation. Constr Build Mater 29:167–174
- Liu Z, Cai C, Liu F, Fan F (2016) Feasibility study of loess stabilization with fly ash-based geopolymer. J Mater Civ Eng 28:0001490
- 39. Arulrajah A, Yaghoubi M, Disfani MM, Horpibulsuk S, Bo MW, Leong M (2018) Evaluation of fly ash-and slag-based

geopolymers for the improvement of a soft marine clay by deep soil mixing. Soils Found 58:1358–1370

- 40. Luo Z, Zhang B, Zou J, Luo B (2022) Sulfate erosion resistance of slag-fly ash based geopolymer stabilized soft soil under semiimmersion condition. Case Stud Constr Mater 17:e01506
- Lorenzo GA, Bergado DT (2006) Fundamental characteristics of cement-admixed clay in deep mixing. J Mater Civ Eng 18:161–174
- 42. Pedarla A, Chittoori S, Puppala AJ (2011) Influence of mineralogy and plasticity index on the stabilization effectiveness of expansive clays. Transp Res Rec 2212:91–99
- Abdullah HH, Shahin MA, Walske ML, Karrech A (2019) Systematic approach to assessing the applicability of fly-ash based geopolymer for clay stabilization. Can Geotech J 57:1356–1368

**Publisher's Note** Springer Nature remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.